

Having thus defined the invention, it is claimed:

1) A system for measuring transmission of a selected gas of interest through a barrier material to establish permeability of said material comprising:

5 a sealable configured unit having first and second facing surfaces confronting one another; one side of said material being positionable adjacent said first surface to form a test gas chamber and the opposite side of said material being positionable adjacent said second facing surface to form a sealed measurement chamber having an area sufficient to permit as a function of the barrier characteristics of said material a
10 uniform diffusion of said gas of interest under a vacuum through said material;

said test gas chamber having an inlet port connected to a source of said selected gas, an outlet port and a flow valve connected to a source of vacuum for controlling flow of said gas into and out of said test gas chamber;

a vacuum system in valved communication with said measurement chamber;

15 a mass spectrometer in fluid communication with said measurement chamber ;

and,

a controller for controlling said mass spectrometer, said vacuum system and said flow valve to permit sampling of said concentration of said gas of interest in said measurement chamber at selected intervals.

2) The system of claim 1 wherein said vacuum system includes (a) a roughing vacuum pump for drawing a vacuum in said test gas chamber, (b) a molecular pump and a second roughing pump for drawing a vacuum in said measurement chamber.

25 3) The system of claim 2 wherein said control means includes a programmed routine implemented by a computer causing sequenced application of said pumps to include an initial application of said roughing vacuum pumps to assure vacuum integrity followed by actuation of said high molecular pump.

4) The system of claim 3 wherein said programmed routine causes said roughing pump of said test gas chamber draws a set vacuum in said test gas chamber while said molecular pump draws a higher set vacuum in said measurement chamber to establish a set pressure differential therebetween depending upon said barrier material composition.

5) The system of claim 2 wherein the area of said measurement chamber is at least about 200 cm² to produce an average number of area defects in said barrier material whereby uniform diffusion of said gas occurs at vacuum levels of at least about 2 times 10⁻⁴ Torr.

6) The system of claim 5 wherein said area is at least about 10 cm².

7) The system of claim 3 wherein said selected gas has an atomic mass lower than about 50 in number.

8) The system of claim 7 wherein said gas is oxygen.

9) The system of claim 7 wherein said gas is water vapor.

10) The system of claim 7 wherein said gas is an inert, elemental gas.

11) The system of claim 7 wherein said gas is helium whereby non-invasive permeability measurements are obtained.

12) The system of claim 2 further including a heater for heating said chambers to set temperatures.

13) The system of claim 3 wherein said sealable configured units are adapted to examine a barrier material including an organic or inorganic substrate having a surface covered by a plurality of barrier coatings whereby transmission of gases through said substrate is inhibited.

14) The system of claim 13 wherein said sealable configured units have a plurality of test gas and measurement sealable chambers generally longitudinally aligned with one another for performing a plurality of simultaneous measurements at set locations intended to measure sheet material.

15) The system of claim 13 further including a payout wheel adjacent one longitudinal end of said box and a take-up reel adjacent the opposite longitudinal end of said box, said computer causing said reels to operate synchronously with one another whereby a sheet roll of said material may be tested.

16) The system of claim 1 further including a porous support for supporting said material against excessive deflection in said measurement chamber.

17) The system of claim 16 wherein said porous support is a grid having a lattice structure lacking sharp edges.

18) The system of claim 1 wherein:

said sealable unit has a continuous seal extending from each facing surface circumscribing a sealable area; a clamp mechanism actuatable from an open position to a clamped position whereat said seal in said first facing surface sealing engages one side of said material to form said test gas chamber extending from said one side of said material and said seal in said second facing surface sealing engages the opposite side of said material to form a sealed measurement chamber extending from said opposite

side of said material; said sealable area bounded by said seal in said second facing surface defining a measurement sealable area spanning a distance sufficient to permit, as a function of the barrier characteristics of said barrier material, a uniform diffusion of said gas under a vacuum through said barrier material extending over said measurement sealable area;

said vacuum system in valved communication with said measurement chamber being sufficient to draw a vacuum of at least about 5×10^{-4} Torr in said measurement chamber when actuated; and,

said mass spectrometer in fluid communication with said measurement chamber being sufficient to analyze the concentration of said gas diffused into said measurement chamber at any given time.

19) A method for continuously measuring the diffusion of a gas through a permeable, barrier material comprising the steps of:

- a) providing a sealable box within which at least a portion of said material is placed;
- b) sealing said material within said box to form a test gas chamber extending from one side of said material and a measurement chamber extending from the opposite side of said material, said measurement chamber encompassing a sealed area of said material sufficient to allow uniform diffusion of said gas through said material;
- c) continuously metering a set quantity of said gas into and out of said test gas chamber by controlling a rough vacuum to determine the concentration of said gas in said test gas chamber as a function of time;
- d) drawing a vacuum in said measurement chamber by a molecular vacuum pump to a final vacuum of at least about 2×10^{-4} Torr;
- e) providing a mass spectrometer in direct valved communication with said measurement chamber; and
- f) determining the transmission of said gas of interest through said material

by partial pressure readings of said mass spectrometer.

20 The method of claim 19 further including the step of initially calibrating said mass spectrometer to a national standard indicative of an absolute permeation measurement
5 to establish an absolute gas transmission rate through said barrier material in step (f).

21 The method of claim 20 further including the step of verifying said measurement chamber against leakage at the final vacuum levels of said measurement chamber.

10 22 The method of claim 21 further including the step of heating said measurement and test gas chambers to a set level whereby the excitation of and the diffusion of said gas into said material is enhanced.

15 23) The method of claim 20 further including the step of providing a roughing vacuum pump valved into fluid communication with said gas and said test gas chamber and said gas is liquid water in a heated, agitated container connected to said roughing pump, said roughing pump causing water vapor to be supplied to said test gas chamber and said mass spectrometer measuring water vapor concentration diffused into said measurement chamber and said correlating step determining the ability of said material
20 to resist permeation of humidity present under normal operating conditions of said material.

24) The method of claim 21 wherein said material includes a substrate with a plurality of barrier coatings applied to a substrate surface and said gas may comprise any
25 elemental gas or combination thereof.

25) The method of claim 24 wherein said gas has an atomic mass lower than about 50 including oxygen and water vapor and combinations thereof.

26) The method of claim 25 wherein said gas is an inert gas including argon, helium and combinations thereof.

5 27) The method of claim 26 wherein said gas is helium and said correlating step includes the step of establishing a relationship between the diffusion rate of helium through said material and the diffusion of a gas of interest through said material.

10 28) The method of claim 20 further including the step of supporting said material against excessive deflection into said measurement chamber.

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29) Apparatus for measuring transmission of at least one gas of interest through plastic barrier materials of the type covering electronic displays including organic light emitting diode displays and field emission displays comprising:

15 a sealable test box having measurement and test gas facing surfaces confronting one another; a continuous seal extending from each facing surface circumscribing a sealable area; a clamp mechanism actuatable from an open position to a clamped position whereat said seal extending from said test gas face surface sealing engages one side of said material to form a test gas sealed chamber extending from said one
20 side of said material and said seal extending from said measurement face surface sealing engages the opposite side of said material to form a measurement sealed chamber extending from said opposite side of said material; said sealable area formed by said seal in said measurement facing surface defining a first sealable area sufficient in size to permit diffusion of said gas through sufficient number of defects in said barrier
25 material to approximate an average gas transmission rate under a hard vacuum through said measurement sealable area;

a roughing vacuum pump in valved fluid communication with said test gas chamber; said test gas chamber having an inlet port connected to a source of said gas,

an outlet port and a flow valve for controlling flow of said gas into and out of said test gas chamber when said roughing vacuum pump is actuated; said roughing vacuum pump in valved fluid communication with said measurement chamber for drawing a rough vacuum in said measurement chamber;

5 a turbomolecular vacuum system in valved communication with said measurement chamber for drawing a vacuum of at least about 2 and 10^{-4} Torr in said measurement chamber when actuated;

a mass spectrometer in valved direct, unimpeded fluid communication with said vacuum system for analyzing the concentration of said gas transmitted into said measurement chamber at any given time; and

10 a computer for controlling through a programmed routine said mass spectrometer, said vacuum pump and said flow valves; said programmed routine effective to initially determine, without the presence of said gas, vacuum integrity of said measurement and test gas chambers at programmed vacuum levels followed by calibration of said mass spectrometer when said turbomolecular system is actuated.

15 30) The apparatus of claim 29 further including a plurality of seals establishing a like plurality of pairs of measurement and test gas chambers longitudinally spaced from one another in said measurement and test gas facing surfaces, each test gas chamber in each pair of chambers having its inlet port in valved fluid communication with a common gas manifold and its outlet port in fluid communication with a common exhaust manifold, said exhaust manifold being valved to an atmospheric vent and to said roughing vacuum pump; said gas manifold in valved fluid communication with a plurality of source gases whereby one or more of said source gases may be valved into fluid
20 communication from said common gas manifold with all of said test gas chambers while the rate of flow of said one or more of said source gases into each test gas chamber being individually set by said computer.

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31) The apparatus of claim 30 wherein each measurement chamber is individually valved to said turbomolecular system whereby pressure differential between each measurement and test gas chamber pairs is controlled.

5 32) The apparatus of claim 31 further including a heater for controllably heating each pair of measurement and test gas chambers.

33) The apparatus of claim 32 further including as a source for one of said gases a container for water, a heater for heating said water to a temperature of approximately 20
10 to 75 degrees C and an agitator to produce water bubbles when said container is subjected to a rough vacuum from said rough vacuum pump whereby water vapor is produced as one of said gases of interest.

15 34) The apparatus of claim 32 wherein said gases of interest have atomic masses lower than about mass 200.

35) The apparatus of claim 34 wherein one of said gases of interest is helium.

20 36) The apparatus of claim 31 wherein said barrier material is in a roll form and said box has a payout reel at one longitudinal end thereof and a take-up reel at the opposite longitudinal end and said programmed routine synchronizing the rotation of said reels to sequentially move said roll longitudinal set distances relative to said box after a plurality of said first and second chambers have analyzed the gas transmission characteristics of said selective gases over a segment of said sheet.

25 37) The apparatus of claim 36 wherein said selective gases of interest are inert gases whereby permeability of said sheet of material may be established by non-contaminating measurements.

38) The apparatus of claim 29 further including a support grid underlying said barrier material in said measurement chamber.

39) A method for determining the transmission characteristics of at least one gas of interest through barrier materials of the type covering electronic displays including organic light emitting diodes and field emission displays comprising the steps of:

a) forming a vacuum barrier across said plastic material by establishing a sealed test gas chamber extending from one side of said plastic material and a sealed measurement chamber extending from the opposite side of said plastic material, said sealed chambers extending over a surface area of said material of at least about 10 cm² whereby sufficient material area is provided to assure average gas transmission characteristics through said material;

b) establishing a rough vacuum across said material between said measurement and test gas chambers;

c) verifying the integrity of said rough vacuum;

d) pumping a high vacuum in said measurement chamber;

e) measuring the background partial pressure of said measurement chamber under said high vacuum;

f) flowing said gas of interest through said test gas chamber under a rough vacuum; and,

g) measuring the resulting change in partial pressure of said measurement chamber over time and correlating the change in partial pressure to the permeability of said material.

40) The method of claim 39 wherein said partial pressure of said measurement chamber is measured by a mass spectrometer having a sensitivity of less than about 2 x 10⁻⁶ Torr.

41) The method of claim 40 further including the step of controlling the temperature of said material to a set level above ambient whereby transmission characteristics of said gas of interest is increased.

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42) The method of claim 40 further including the step of controlling the vacuum on each side of said material in said measurement and test gas chambers to a set level whereby pressure across said material is reduced to avoid material distortion and/or failure.

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43) The method of claim 40 further including the step of controlling the gas flow into said test gas chamber to regulate the gas pressure across said material.

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44) The method of claim 40 further including the step of providing an array of pairs of measurement and test gas chambers extending in a longitudinal direction; providing said material as a strip wound on a pay-out reel at one longitudinal end of said array and rewound on a take-up reel at the opposite longitudinal end of said array and said gas of interest being an inert gas whereby non-invasive permeability measurements may be taken by sequentially measuring transmission characteristics of said inert gas through said sheet material.

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45) The method of claim 40 further including the step of physically supporting said barrier material against deflection into said measurement chamber.

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46) A system for measuring transmission of a gas or gases of interest through a barrier material comprising:

a test unit having a test gas chamber extending from one side of said barrier material and a sealed measurement chamber extending from the opposite side of said barrier material;

a vacuum system in continuous valved communication with i) said test gas chamber for continuously drawing said gas of interest through said test gas chamber and ii) said measurement chamber for continuously drawing said gas of interest from said measurement chamber; and,

5 a residual gas analyzer for analyzing said gas of interest drawn from said measurement chamber.

47) The system of claim 46 wherein said vacuum system includes a roughing pump for drawing a rough vacuum in said test gas chamber and a molecular pump for drawing
10 a high vacuum in said measurement chamber.

48) The system of claim 46 wherein said residual gas analyzer is a mass spectrometer.

49) The system of claim 46 wherein said gas of interest is helium.

50) A method for measuring transmission of a gas of interest through a barrier material comprising the steps of:

a) providing a test gas chamber in fluid communication with a measurement chamber but for the presence of said barrier material therebetween;

20 b) drawing a first vacuum in said test gas chamber and a lower pressure second vacuum in said measurement chamber so that a pressure differential exists therebetween;

c) continuously metering said gas of interest to said test gas chamber and analyzing at any selected time the concentration of said gas of interest in said
25 measurement chamber.

51) The method of claim 50 further including the step of providing a mass spectrometer and analyzing said gas of interest present in said measurement chamber

at selected time increments by said mass spectrometer.

52) The method of claim 50 further including the steps of:

establishing, for any given gas of interest and any given barrier material, a
5 correlation between the concentration of said gas of interest passing through an
unsaturated barrier material until said barrier material becomes saturated with said gas
of interest and the time expended before said unsaturated barrier material becomes
saturated;

10 testing a barrier material with said given gas of interest by determining the
concentration of said given gas of interest passing through said barrier material for a
second time period less than that required to achieve saturation of said barrier material
with said given gas of interest; and,

15 comparing the concentrations of said given gas obtained from said tested barrier
material with said correlated concentration obtained during said second interval to
determine acceptance or rejection of said tested barrier material.

53) The method of claim 50 further including the steps of using helium as said gas of
interest and correlating the concentrations of helium gas transmitted through a given
barrier material to said measurement chamber as a function of time with the
20 concentration of a given gas of interest transmitted through said given barrier material
as a function of time.

54) The method of claim 53 wherein said time extends to a time period whereat
saturation of said helium gas with said given barrier material and saturation of said
25 given gas of interest with said given barrier material occurs.

55) The method of claim 53 wherein said time for correlating said gas concentration
is a fixed time.

56) A method for determining the gas transmission characteristics of a given gas of interest passing through a given barrier material comprising the steps of:

a) measuring the gas transmission characteristics of helium passing through said given barrier material; and,

b) correlating the measured helium gas transmission characteristics with said given gas of interest.

57) The method of claim 56 wherein the step of measuring is determined by establishing a pressure differential between helium on one side of said given barrier material with a vacuum on the other side of said given barrier material, said pressure differential effective to draw said helium through said given barrier material.

58) The method of claim 57 wherein a mass spectrometer measures the partial pressure of said helium present in said vacuum.